

## UNITED STATES PATENT OFFICE

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## TUNGSTEN MANUFACTURE

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This invention relates to refractory metal manufacture. More specifically, the invention pertains to a refractory metal such as tungsten in coiled filamentary form and a process for making the same. The invention has particular application to electric lamps.

In the use of refractory metals such as tungsten in filamentary form for electric lamps difficulties have arisen from the fact that at the high temperatures at which these lamps are operated the filaments are subjected to structural changes which cause "offsetting" and "sagging". By "offsetting" is meant a faulting or bodily shifting of the material of the filament, causing a break in the filament. This is believed to be due to the gradual crystallization of the metal along a plane which so weakens the strength of the material as to permit lateral or side slipping or shifting between the grain surfaces and final separation of the material. By the term "sagging" is meant the gradual displacement of the material causing a lengthening of the filament between its supports and consequent deformation of the coil. This is disadvantageous, as the sagging modifies the electrical and light-giving characteristics of the filament disadvantageously and in a manner not easily predictable from the original lamp filament dimensions.

Various means have been proposed to overcome these difficulties in lamp filaments, such as the formation of the metal into a single elongated crystal, or the introduction into the filament during the process of manufacture of certain foreign substances such as thoria or silica, which supposedly have an effect upon the crystalline growth of the tungsten in such manner as to tend to resist the development of offsetting or sagging. These various methods, while useful, have not been effective to completely overcome the mentioned difficulties.

Accordingly, it is one of the important objects of the present invention to provide means for obtaining improved results as regards sagging and offsetting in refractory filaments for electric lamps. A further object is to provide a new type of filament which gives increased efficiency in electric lamp operation. Further objects of the invention relate to improved manufacturing methods and to the production of a metal filament which retains, after high temperature heating, its original elasticity. Various other objects will appear from the following description of my invention, which I will describe with particular reference to the use of tungsten metal in filamentary form for electric lamps.

As a primary source of the tungsten employed in the filament I use tungstic acid ( $\text{H}_2\text{WO}_4$ ) in powdered form and in a chemically pure state. This acid is mixed preferably with potassium fluotitanate ( $\text{K}_2\text{TiF}_6 \cdot \text{H}_2\text{O}$ ), although other titanium compounds such as titanium fluoride or titanium potassium oxalate may be used. In combining these substances the fluotitanate is first dissolved in water and the powdered tungstic acid then added. This mixture is mechanically stirred over a steam bath until the water is evaporated to dryness, and the resulting material is then crushed and loaded in fused silica crucibles and heated to a temperature somewhere in the neighborhood of  $1,000^\circ \text{C}$ . to bring about dehydration of the constituent compounds. Alternately the crucible heating may be omitted as not essential, the dehydration being accomplished as a preliminary phase of the subsequent reduction heating.

The resulting product of the crucible heating is then pulverized and loaded into boats in a reduction furnace where it is subjected to a continuous heating for approximately three and one-third hours to bring about the reduction of the tungstic anhydride ( $\text{WO}_3$ ) and the initial development of the crystalline or grain structure of the tungsten. I employ a type of oven wherein nichrome tubing is heated externally by gas flames in at least three sections of more or less uniform temperature, the first section being maintained at approximately  $388^\circ \text{C}$ ., the second at  $833^\circ \text{C}$ ., and the third at  $1,000^\circ \text{C}$ . The stoking time varies from ten to twenty-five minutes, and inasmuch as there are ten boats in the furnace at a time, it is apparent that each boat receives progressively and uniformly the temperature of each heated section. While these boats are being passed through the tubing, hydrogen gas is drawn through the tubing in a direction opposite to the movement of the boats in a quantity depending upon the speed of movement and the amount of charge of the boats through the furnace, an average flow being approximately 100 cubic feet per minute. The speed of movement of the boats and the rate of hydrogen flow and amount of charge are all important as affecting the grain size of the resultant tungsten; the faster the movement for a maintained rate of hydrogen flow the larger the crystalline size. Conversely provided the size of charge remains the same, an increased flow of hydrogen with the stoking rate remaining the same results in decreasing, or a maintaining of the hydrogen rate at a constant value and a